

# ***U.S. PATENT APPLICATION***

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***Invention:*** DEVICE AND METHOD FOR DETECTING SULFURYL FLUORIDE

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## ***SPECIFICATION***

## DEVICE AND METHOD FOR DETECTING SULFURYL FLUORIDE

### CROSS REFERENCE TO RELATED APPLICATION

[0001] This application is based on and incorporates herein by reference German Patent Application No. DE 102 54 748.3-52, filed on November 23, 2002.

### BACKGROUND OF THE INVENTION

[0002] The invention relates to a device for detecting sulfuryl fluoride and to a method for detecting sulfuryl fluoride.

[0003] Sulfuryl fluoride ( $\text{SO}_2\text{F}_2$ ) is for instance used as a fumigant for killing insects. Sealed rooms in a building are fumigated, using a predetermined action time. The measurement task is on the one hand to trace leaks by way of which the fumigant escapes, and to perform a free measurement after the conclusion of the fumigation.

[0004] For detecting sulfuryl fluoride, direct-indicating measuring instruments are known in which  $\text{SO}_2\text{F}_2$  is decomposed in a pyrolysis oven, and the resultant sulfur dioxide is detected with a measurement cell. The detection limit is in a range between 0 and 50 ppm. One such measuring instrument is offered by the company doing business as Ansyco, Analytische Systeme und Komponenten GmbH, with the product designation "GF 1900". Although with the known measuring instrument very low concentrations can be detected, the electrically operated pyrolysis oven requires a heavy battery pack, making the

measuring instrument, particularly for leakage measurement, only conditionally portable. The pyrolysis oven furthermore requires major expense for apparatus.

[0005] For detecting chlorinated hydrocarbons, it is indeed known from European Patent Disclosure EP 281 938 A1 first to subject the gas specimen to be examined to pyrolysis and then to detect the pyrolysis product, in this case a chlorine compound, with a test tube, but in the known prior art no information can be found on detecting sulfuryl fluoride.

#### BRIEF DESCRIPTION OF THE INVENTION

[0006] The object of the invention is provide a device and a method for mobile, economical detection of sulfuryl fluoride.

[0007] This object is attained with a device for detecting sulfuryl fluoride, in which the gas specimen to be examined is subjected to pyrolysis, with ensuing detection of a pyrolysis product, wherein for the pyrolysis, a chemical layer of pyrophoric iron is provided, and that as an indication system for the pyrolysis product, a test tube for hydrogen fluoride is present.

[0008] For the method, this object is attained with a method for detecting sulfuryl fluoride, in which the gas specimen to be examined is subjected to pyrolysis, with ensuing detection of a pyrolysis product, wherein the pyrolysis is performed with a chemical layer of pyrophoric iron above 400° Celsius, and the hydrogen

fluoride concentration of the pyrolysis product is detected, using a colorimetric test tube.

[0009] The advantage of the invention is essentially to perform the pyrolysis of  $\text{SO}_2\text{F}_2$  with a test tube filled with chemicals that is disposed as a preliminary tube on the oncoming-flow side of a commercially available test tube for detecting hydrogen fluoride. The test tube for detecting hydrogen fluoride can be procured for instance from Dräger Safety AG & Co. KGaA, with the product designation "HF 1,5/b- Röhrchen" (HF 1.5/b tube). It is expedient to provide a scale for sulfuryl fluoride, so that a direct readout is possible.

[0010] The preliminary tube contains finely divided pyrophoric iron, which because of its large surface area and lattice flaws spontaneously ignites in air and thus attains high temperatures of over 400° Celsius. Pyrophoric iron is produced by the decomposition of iron oxalate; depending upon the iron oxalate quantity used, approximately 25% pyrophoric iron is created, the rest being iron oxide. In the pyrolysis, the pyrophoric iron is converted into solid iron oxide, and sulfuryl fluoride is split into hydrogen fluoride and sulfur dioxide.

[0011] Advantageous features of the invention will become apparent from the dependent claims.

[0012] Advantageously, the pyrolysis can be markedly improved, and the detection limit of sulfuryl fluoride thus markedly lowered, by adding iron powder or aluminum powder to the pyrophoric iron. This creates more

combustible material, and the pyrolysis is optimized in terms of detecting small concentrations of sulfuryl fluoride.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0013] The sole drawing figure is a schematic view of the device of the invention.

#### DETAILED DESCRIPTION OF THE INVENTION

[0014] One exemplary embodiment of the invention is shown in the drawing and described in further detail below.

[0015] The sole drawing figure shows the device of the invention with a preliminary tube 1 and a colorimetric test tube 2 for hydrogen fluoride, of the kind offered by Dräger Safety AG & Co. KGaA under the designation "Fluorwasserstoff 1,5 b, Sachnummer CH 30 301" (Hydrogen fluoride 1.5 b, item number CH 30 301). The tubes 1, 2 are joined together by a hose 3, and the flow direction is represented by arrows 4. The preliminary tube 1 contains a chemical layer 5 between two retaining elements 6, 7 and also contains two quartz glass granulate layers 8, 9. The quartz glass granulate layers 8, 9 serve to prevent the chemical layer 5 from trickling through the retaining elements 6, 7. In the chemical layer 5, iron oxalate and very fine iron powder are mixed together. After the filling, the chemical layer 5 in the preliminary tube 1 is heated to over 400° Celsius. In the process, iron oxalate is converted into pyrophoric iron and iron oxide. The proportion of iron, which reacts

strongly exothermally with oxygen when the specimen is taken, is enhanced by the addition of iron powder. Pyrophoric iron is distinguished from normal iron powder by flaws in the lattice. These flaws, together with the large surface area of the finally divided iron, assure that the iron will react so strongly with oxygen that bright red heat results. As the pyrophoric iron is burned off, no combustion gases occur. The colorimetric test tube 2 contains an indicator layer 10 of zirconium quinalizarine and is provided with a scale 11 for the hydrogen fluoride concentration, with a measurement range from 1.5 ppm to 15 ppm.

[0016] While the invention has been described in connection with what is presently considered to be the most practical and preferred embodiment, it is to be understood that the invention is not to be limited to the disclosed embodiment, but on the contrary, is intended to cover various modifications and equivalent arrangements included within the spirit and scope of the appended claims.